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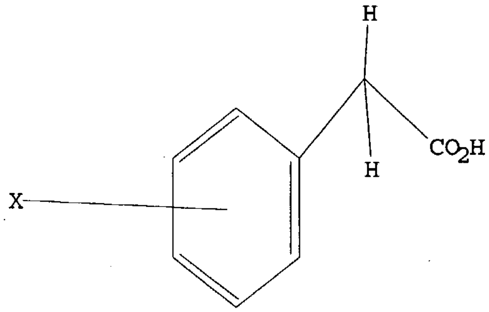

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Structure attributes must be viewed using STN Express query preparation.


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Substance data SEARCH and crossover from CAS REGISTRY in progress...
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FULL SEARCH INITIATED 18:43:37 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 115162 TO ITERATE

100.0% PROCESSED  115162 ITERATIONS                          2654 ANSWERS
SEARCH TIME: 00.00.01

L2          2654 SEA SSS FUL L1



L3         4983 L2

=> s l3 and decarboxylat?
         35993 DECARBOXYLAT?
L4           152 L3 AND DECARBOXYLAT?

=> s l4 and dicarboxy?
         76302 DICARBOXY?
L5             4 L4 AND DICARBOXY?

=> d 1-4 ibib abs hitstr

L5   ANSWER 1 OF 4  CAPLUS  COPYRIGHT 2004 ACS on STN
ACCESSION NUMBER:         1984:551754  CAPLUS
DOCUMENT NUMBER:          101:151754
TITLE:                    Phenol derivatives
INVENTOR(S):              Wenk, Paul; Breitenstein, Werner; Baumann, Marcus
PATENT ASSIGNEE(S):       Ciba-Geigy A.-G. , Switz.
SOURCE:                   Eur. Pat. Appl., 96 pp.
                          CODEN: EPXXDW
DOCUMENT TYPE:            Patent
LANGUAGE:                 German
FAMILY ACC. NUM. COUNT:   1
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PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
EP 107620	A1	19840502	EP 1983-810462		19831007
R: AT, BE, CH, DE, FR, GB, IT, LI, LU, NL, SE					
FI 8303676	A	19840414	FI 1983-3676		19831010
DK 8304706	A	19840414	DK 1983-4706		19831012
NO 8303709	A	19840416	NO 1983-3709		19831012
AU 8320114	A1	19840419	AU 1983-20114		19831012
JP 59093021	A2	19840529	JP 1983-189430		19831012
DD 218348	A5	19850206	DD 1983-255614		19831012
ZA 8307572	A	19850424	ZA 1983-7572		19831012
HU 35243	O	19850628	HU 1983-3524		19831012
PRIORITY APPLN. INFO.:			CH 1982-5987	A	19821013

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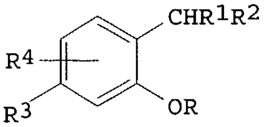

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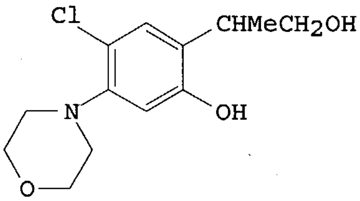


II

AB Phenol derivs. I (R = H, acyl; R1 = CH2OH, esterified or etherified CH2OH, CHO, acetalized CHO; R2 = H, aliphatic group; R3 = NH2 disubstituted by hydrocarbyl or hydrocarbondiyl optionally with hetero interrupter; R4 = H, substituent), useful as inflammation inhibitors, antipyretics, analgesics (no data), and as sunscreens selectively blocking 290-320 nm burning radiation while passing browning rays of .apprx.320-400 nm, were prepared Morpholinophenol II was prepared in 5 steps from imidazo[1,2-a]pyridin-2(3H)-one hydrochloride, NaOH, and maleic acid via 4-methyl-3-(3-oxobutyl)maleic anhydride and 3-methyl-6-morpholinobenzofuran-2(3H)-one.

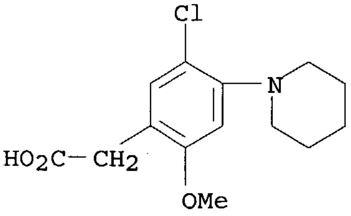
IT **87203-09-6P**

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and lithium aluminum hydride reduction of)

RN 87203-09-6 CAPLUS

CN Benzeneacetic acid, 5-chloro-2-methoxy-4-(1-piperidinyl)- (9CI) (CA INDEX NAME)



L5 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1983:539751 CAPLUS
DOCUMENT NUMBER: 99:139751
TITLE: Furans
INVENTOR(S): Wenk, Paul; Breitenstein, Werner; Baumann, Marcus
PATENT ASSIGNEE(S): Ciba-Geigy A.-G. , Switz.
SOURCE: Eur. Pat. Appl., 103 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
EP 78241	A2	19830504	EP 1982-810439		19821022
EP 78241	A3	19840328			
R: AT, BE, CH, DE, FR, IT, LI, LU, NL, SE					
US 4426380	A	19840117	US 1982-435595		19821021
FI 8203640	A	19830429	FI 1982-3640		19821025
GB 2110210	A1	19830615	GB 1982-30351		19821025
GB 2110210	B2	19850703			
ES 516842	A1	19840116	ES 1982-516842		19821026
CA 1199635	A1	19860121	CA 1982-414197		19821026
DK 8204759	A	19830429	DK 1982-4759		19821027
NO 8203585	A	19830429	NO 1982-3585		19821027
AU 8289823	A1	19830505	AU 1982-89823		19821027
ZA 8207844	A	19830629	ZA 1982-7844		19821027
DD 204699	A5	19831207	DD 1982-244314		19821027
HU 29609	O	19840228	HU 1982-3447		19821027
JP 58126882	A2	19830728	JP 1982-191737		19821028
US 4451462	A	19840529	US 1983-542334		19831017
ES 526890	A1	19851001	ES 1983-526890		19831028
ES 526892	A1	19851001	ES 1983-526892		19831028
ES 526891	A1	19860201	ES 1983-526891		19831028
PRIORITY APPLN. INFO.:			CH 1981-6882	A	19811028
			US 1982-435595	A1	19821021

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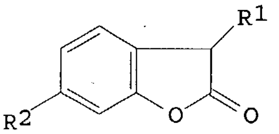

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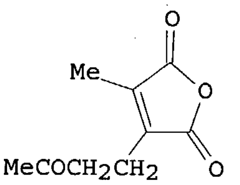


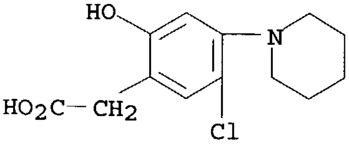
II

AB Benzofuranones I (R1 = H, aliphatic group; R2 = amino disubstituted with hydrocarbondiyl; benzo ring may be addnl. substituted) and their salts and(or) isomers, useful as inflammation inhibitors, analgesics, and sunscreens for skin (no data), were prepared Imidazo[1,2-a]pyridin-2(3H)-one hydrochloride in aqueous NaOH added to maleic acid to give 3-(1,2-**dicarboxyethyl**)imidazo[1,2-a]pyridin-2-(3H)-one. The HCl salt of this added to MeCOCH:CH2 and the product was **decarboxylated** and hydrolyzed to give maleic anhydride II. This reacted with morpholinium benzoate in refluxing C6H6 in 48 h with H2O separation to give I (R1 = Me, R2 = morpholino).

IT **87202-99-1P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and cyclization of)

RN 87202-99-1 CAPLUS

CN Benzeneacetic acid, 5-chloro-2-hydroxy-4-(1-piperidinyl)- (9CI) (CA INDEX NAME)

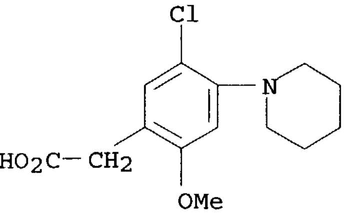


IT **87203-09-6P 87203-10-9P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and ether cleavage of)

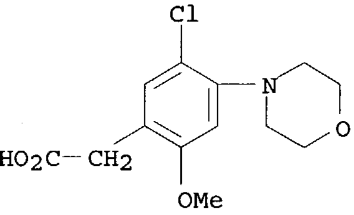
RN 87203-09-6 CAPLUS

CN Benzeneacetic acid, 5-chloro-2-methoxy-4-(1-piperidinyl)- (9CI) (CA INDEX

NAME)



RN 87203-10-9 CAPLUS
CN Benzeneacetic acid, 5-chloro-2-methoxy-4-(4-morpholinyl)- (9CI) (CA INDEX NAME)



L5 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1960:118369 CAPLUS
DOCUMENT NUMBER: 54:118369
ORIGINAL REFERENCE NO.: 54:22677e-i,22678a-i,22679a
TITLE: Nucleophilic aromatic substitutions. XVIII. New ring closures through arynes
AUTHOR(S): Huisgen, Rolf; Konig, Horst; Lepley, Arthur R.
CORPORATE SOURCE: Univ. Munich, Germany
SOURCE: Chemische Berichte (1960), 93, 1496-1506
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 54:118369

AB cf. CA 54, 19618a. The intramol. addition of a secondary amino group to arynes proved to be useful for the closure of large rings. In the cyclization of methyl-[ω(2,5-dichlorophenyl)alkyl]amines with PhLi, elimination of the Cl in m-position to the side chain was preferred. The intramol. addition of alcoholate to aryne can be accomplished, but the similar intermol. addition was observed. Methyl[12-(m-chlorophenyl)dodecyl]amine (I) (1.45 g.) in 50 cc. dry C6H6 added dropwise during 150 hrs. to 5.0 g. NaNH2 in 450 cc. boiling C6H6, the aqueous phase extracted with Et2O, and the combined organic solns. distilled yielded 1.21 g. oil, b0.005 130-205°; a 1.00-g. portion refluxed with Ac2O in C6H6 and evaporated, and the residue chromatographed on 150 g. Al2O3 yielded 607 mg. mixture of 4-methyl-4-aza-1,3-benzocyclohexadecene (II) and 5-methyl-5-aza-1,4-benzocycloheptadeca-1,3-triene (III) and 380 mg. of the N-Ac derivative of I and of Ph(CH2)12 NHMe. The II-III mixture treated with styphnic acid gave the styphnate (IV) of II, pale yellow rodlets, m. 144.5-5.3° (loss of solvent can be observed under the microscope at 120°). IV decomposed with NH4OH yielded pure II, b0.005 125-30° (bath), m. 32.5-33°. II-III mixture treated with alc. picric acid yielded the picrate (V) of III, light yellow leaflets, m. 186-7° (decomposition) (EtOH-C6H6). I (20.6 mmoles) added during 112 hrs. to 5 g. NaNH2 in 750 cc. refluxing PhMe and worked up in the usual manner gave 38% II and 4% III. I (30 mmoles) treated during 105 hrs. with 160 mmoles PhLi in 3000 cc. Et2O by the method described previously (CA 53, 11401b) gave 10% unchanged I, 29% II, and 3% III. A similar run during 160 hrs. with 30 mmoles I and 120 mmoles PhLi yielded 20% unchanged I, 35% II, and 3% III. 2,5-Cl2C6H3Me chlorinated with SO2Cl2 and Bz2O2 yielded 2,5-Cl2C6H3CH2Cl (VI), b10 102°. VI (93 g.) treated with

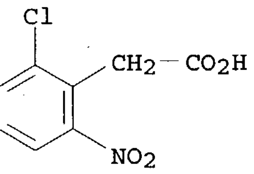
13 g. Na and 100 cc. CH2(CO2Et)2 in absolute EtOH gave 109 g. 2,5-Cl2C6H3CH2CH(CO2Et)2, b0.005 123-40°; a 208-g. batch saponified with NaOH-MeOH and acidified, and the precipitated **dicarboxylic** acid **decarboxylated** at 140° yielded 131 g. 2,5Cl2C6H3(CH2)2CO2H (VII), m. 96-7° (cyclohexane and then H2O). VII oxidized with KMnO4 gave 2,5-Cl2C6H3CO2H, m. 153-4°. VII degraded by the method described previously (CA 53, 10205i) gave 67% 2,5-Cl2C6H3(CH2)2NH2 (VIII), b10 132-4°, n20D 1.5680; 2VIII.(CO2H)2, m. 220-1° (decomposition). VIII (33 g.) and 190 g. HCO2Et refluxed to solution and distilled yielded 38 g. 2,5-Cl2C6H3(CH2)2NHCHO (IX), m. 74-5° (EtOH-cyclohexane). IX reduced with LiAlH4 yielded 57% 2,5-Cl2C6H3(CH2)2NHMe (X), b15 140°; X.(CO2H)2, m. 203-4° (decomposition) (MeOH-EtOAc). 2,5-Cl2C6H3(CH2)2CO2Et reduced with LiAlH4 and the resulting carbinol treated with HBr gave 2,5-Cl2C6H3(CH2)3Br, b10 155-7°, n20D 1.5730, which heated 20 hrs. at 100° with excess MeNH2 in a sealed tube gave 75% 2,5-Cl2C6H3(CH2)3NHMe (XI), b10 148-50°, n25D 1.5418; XI.(CO2H)2, needles, m. 196-7° (decomposition) (MeOH); picrate m. 140-1° (EtOH). 2,6-Cl(O2N)C6H3CH2Br (34 g.) and 33 g. KCN in 40 cc. H2O and 150 cc. EtOH refluxed 1 hr. with stirring, diluted with H2O, and extracted with Et2O, and the residue from the extract extracted with 1:4 C6H6-petr. ether gave 24 g. 2,6-Cl(O2N)C6H3CH2CN (XII), m. 67-8° (MeOH). XII hydrolyzed with AcOH-H2O-H2SO4 yielded 87% 2,6-Cl-(O2N)C6H3CH2CO2H (XIII), m. 187-9°, which with MeOH-H2SO4 yielded the Me ester (XIV) of XIII, m. 51-1.5°. XIV hydrogenated in MeOH under ambient conditions over Raney Ni yielded 70% 6-NH2 analog (XV), m. 54-5°, which boiled in MeOH was converted to 4-chlorooxindole; the crude XV in Et2O reduced immediately with LiAlH4 gave 2,6-H2NClC6H3(CH2)2OH (XVI), m. 82-3° (C6H6-petr. ether). XVI cyclized at 210-15° with ZnCl2 yielded 72% 4-chloroindoline (XVII), b10 135°, n25D 1.6034; picrate, light yellow needles, m. 182-3°. XVII treated 12 hrs. at room temperature with pure HCO2H gave the N-CHO derivative, needles, m. 90-1° (cyclohexane), which reduced 6 hrs. with LiAlH4 in refluxing Et2O yielded 82% N-Me derivative (XVIII) of XVII, needles, m. 90-1° (cyclohexane); picrate m. 149-50° (decomposition). p-ClC6H4NHMe was converted in C5H5N in nearly quant. yield into the N-ClCH2CO derivative, m. 168°, and this heated 4 hrs. with twice the amount of AlCl3 at 225° under N, poured onto ice, and extracted with C6H6 yielded 79% 1-methyl-5-chlorooxindole (XIX), b0.01 130°, m. 104-5° (cyclohexane). XIX reduced with LiAlH4 gave a poor yield of 1-methyl-5-chloroindoline (XX), b14 128-9°; picrate m. 169° (EtOH). 1-Methyl-5-chloroquinolinium iodide, m. 190° (decomposition), reduced with Sn and HCl, and the resulting mixture fractionated gave N-methyltetrahydroquinoline and N-methyl-5-chloro-1,2,3,4-tetrahydroquinoline (XXI), b12 152°, n25D 1.5937; picrate m. 130-1° (decomposition) (EtOH). 6-Chloroquinoline-MeI was converted similarly to the 6-Cl isomer (XXII) of XXI, b12 158-60°; picrate m. 127-8° (EtOH). X (2.03 g.) and 20 mmoles PhLi in 400 cc. Et2O refluxed 50 hrs. and hydrolyzed gave 1.67 g. mixture of 67% XVIII and 12% XX, which with picric acid in EtOH yielded the picrate of XVIII, m. 149-50°. A series of similar runs with 10 mmoles X in 400 cc. refluxing Et2O was performed (mmoles PhLi used, reaction time in hrs., and % yields of X, XVIII, and XX given): 11, 10, 35, 27, 10; 15, 96, 11, 44, 13; 18 (and 12 mmoles LiNEt2), 16, 0, 53, 11; 30, 16, 0, 57, 9 XI (3.27 g.) and 25 mmoles PhLi in 500 cc. Et2O refluxed 18 hrs. and worked up in the usual manner yielded 1.50 g. mixture containing 790 mg. XI, 420 mg. XXI, and 147 mg. XXII. m-ClC6H4CH2CO2Me, b9 120-1°, n20D 1.5238, reduced with LiAlH4 yielded 88% m-ClC6H4(CH2)2OH (XXIII), b9 125-8°, n20D 1.5491; phenylurethan derivative, needles, m. 74-5° (petr. ether); p-nitrobenzoate, leaflets, m. 64-5°. m-ClC6H4(CH2)3OH (XXIV) was converted to the p-nitrobenzoate, pale yellow leaflets, m. 39-40° (EtOH). XXIII (6.28 g.) and 4.38 g. Et2NH in 600 cc. Et2O refluxed 50 hrs. with 100 mmoles PhLi, hydrolyzed, and worked up in the usual manner gave 2.83 g. oil, b10 70-129%, containing 28% XXIII and 6.3% coumaran (XXV); the oil washed with alkali and redistd. gave an oil, b10 65-75° (bath), n20D 1.5442, which treated with alc. picric acid yielded the picrate of XXV, m. 75-6° (CHCl3); the neutral fraction, b10 85-120°, contained in addition to XXIII some Ph2. XXV (20 mmoles) and 30 mmoles Et2NLi in 400 cc. Et2O refluxed 45 hrs. and hydrolyzed gave 0.61 g. o-CH2CHC6H4OH, b10 80-90°-(bath), n20D 1.5851 [identified as

2,4,6-Br2(BrCH2CHBr)-C6H2OH, m. 104-5° (C6H6), and as o-HO2CCH2OC6H4CH:CH2, m. 137-8°], and 0.26 g. higher-mol.-weight residue. Et2NLi (30 mmoles) in 225 cc. Et2O added during 30 hrs. to 20 mmoles Li derivative of XXIV in 400 cc. Et2O, refluxed 37 hrs., hydrolyzed and worked up yielded 2.56 g. product containing 56% XXIV and 1.0% chroman (XXVI). PhO(CH2)3OH cyclized with ZnCl2 gave XXVI, b10 86-7°, n20D 1.538. The basic product, b10 163-73°, consisted possibly of a mixture of 1-hydroxy-3-(o- and m-diethylaminophenyl)-1-propene. XXIV (20 mmoles) treated with 30 mmoles Et2NH, 50 mmoles PhLi, and 300 cc. Et2O during 45 hrs. gave 42% unchanged XXIV and 0.6% XXVI. XXIV (48 mmoles) in 250 cc. C6H6 added during 60 hrs. to 10 g. NaNH2 in 300 cc. refluxing C6H6, refluxed 15 hrs., and worked up in the usual manner yielded 0.2% XXVI.

IT **31912-08-0**, Acetic acid, (2-chloro-6-nitrophenyl)-
(preparation of)

RN 31912-08-0 CAPLUS

CN Benzeneacetic acid, 2-chloro-6-nitro- (9CI) (CA INDEX NAME)



L5 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1959:121832 CAPLUS
DOCUMENT NUMBER: 53:121832
ORIGINAL REFERENCE NO.: 53:21808e-i,21809a-i,21810a-b
TITLE: Reactions of methyl and ethoxy free radicals in chlorohydrocarbons: A comparative study of the use of diacetyl peroxide and diethyl peroxydicarbonate as agents for linking alpha carbon to alpha carbon in some chloro-substituted aralkyls
AUTHOR(S): McBay, Henry C.; Tucker, Ozie; Groves, Paul T.
CORPORATE SOURCE: Morehouse Coll., Atlanta, GA
SOURCE: Journal of Organic Chemistry (1959), 24, 536-45
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB Ac2O2 (I) reacts with approx. equal facility with the chloro-substituted aralkyls, 3,4-dichlorotoluene (II), 2,6-dichlorotoluene (III), and α,α-dichlorotoluene (benzal chloride) (IV) to give the corresponding chloro-substituted bibenzyls derived from the dimerizations at the alpha positions. Diethyl peroxydicarbonate (V) reacts with II in the same manner as does I to produce 1,2-bis(3,4-dichlorophenyl)ethane (VI), in essentially equal yield. V gives a poorer yield of the same dimer, 2,2',6,6'-tetrachlorobibenzyl (VII), as obtained from the reaction of I with III. Practically none of the dimer, tetrachlorotolan (VIII), obtained from the reaction of IV with I, is produced when V is used as the linking agent. I links p-isopropylbenzal chloride (IX) unsymmetrically with itself to produce 1,1-dichloro-1-(p-isopropylphenyl)-2-methyl-2-(ω,ω-dichloro-p-tolyl)propane (X) exclusively, while V links IX symmetrically with itself to produce exclusively 2.3-bis(ω,ω-dichloro-p-tolyl)-2,3-dimethylbutane (XI). I and V were prepared, purified, and analyzed by known methods. III b10 72.5°, n20D 1.5517; II b14 85.5°, n20D 1.5490; IV b4.5 45.5°, n20D 1.5509. p-Isopropylbenzaldehyde with PCl5 gave IX, b2 104-6°, n20D 1.5340. I (45.9 g.) in 296.7 g. IV was introduced slowly beneath the surface of 152.1 g. IV held at 115°. From the thermal decomposition of the peroxide there were obtained 27.5 g. CO2, 0.45 g. HCl, 0.2 g. MeOAc, and 12.2 l. CH4. Distillation of the nonvolatile contents gave 366.1 g. unreacted IV and 74.9 g. of crystals in the stillpot, which triturated with Norit in hot ligroine and recrystd. gave 1,2-diphenyl-1,1,2,2-tetrachloroethane, m. 161-2° (ligroine). V (0.49 mole) in 283.2 g. IV slowly added to 97.3 g. IV at 92-5°, gave the following volatile products: 36.5 g. CO2, 1 g. HCl, 29.8 g. alc., and 6.6 g. AcH. The material remaining in the distilling flask gave 295.2 g. IV and 55.8 g. of oil residue which deposited crystals of VIII, m. 162-3°. I (34 g.) in 175 ml. III added dropwise at 130-40° to 30.2 g. III gave the following volatile products: 20.2 g. CO2, 0.2 g. HCl, 3.7 g. MeOAc, and 8.51.CH4. The residue on distillation gave 158.2 g. unreacted III and the residue deposited 38.2 g. VII, b12 76°, m. 155°. All attempts to oxidize VII to 2,6-dichlorobenzoic acid failed. KMnO4, CrO3, and K2Cr2O7 were used as oxidizing agents. Prolonged refluxing (92 hrs.) of 1 g. VII in 50 ml. 50% concentrated HNO3-H2SO4 gave a solution which upon dilution with H2O gave crystals, m. 295°. No attempt was made to determine the positions of the NO2 groups in this tetranitro derivative V (0.38 mole) in 249 g. III added dropwise at 110° to 132.2 g. III gave 32.9 g. CO2, 29.9 g. alc., and 4.2 g. AcH. Unchanged III (347 g.) was recovered and the residue gave 25.8 g. VII. III chlorinated with SO2Cl2 and Bz2O2 gave 60% 2,6-dichlorobenzyl chloride (XII), b3 85-8°, m. 49-50°. XII (20 g.) added in Et2O to 2.5 g. Mg, and after complete reaction dry CO2 gas passed into the system gave 60% 2,6-dichlorophenylacetic acid, m. 156° (aqueous alc.); phenacyl ester m. 60-2° (95% alc.). XII (20 g.) in Et2O converted into the Grignard reagent, and the system refluxed 10 hrs., the solution poured off, and the salt extracted with more Et2O, and the Et2O evaporated gave an oil which deposited crystals, which on treatment with Norit gave 30% VII. Despite the failure to oxidize this compound to known degradation products, the structure seems well established. I (32.5 g.) in 347.5 g. II added dropwise at 130° to 105.7 g. II gave 17.9 g. CO2, 4.7 g. MeOAc, and 6.01 l. CH4. The residue gave 420 g. unchanged II and 28.9 g. VI, m. 111° (95% alc.). V (0.36 mole) in 516 g. II added dropwise at 120° to 70.5 g. II gave the following products: 26.5 g. CO2, 28.8 g. alc., and 1.6 g. AcH. The residue gave 501 g. unchanged II and 63.2 g. crude VI; in equal amts. these m. 111°. VI (2 g.) was oxidized quant. to 3,4-dichlorobenzoic acid, m. 205.5-6.0°. II was chlorinated with SO2Cl2 and Bz2O2 to give 60% 3,4-dichlorobenzyl chloride (XIII), b1.5 84°, b5 104°, n22D 1.5763. XIII was converted to the corresponding Et benzyl ether to demonstrate that the 3rd Cl had replaced a H in the aliphatic portion of the mol. XIII (87.6 g.) refluxed 1.5 hrs. in 300 ml. 60% aqueous alc. containing 22.5 g. NaOH and cooled gave 2 layers; the top layer contained very little of the product, and the bottom layer on evaporation gave 65.2 g. Et 3,4-dichlorobenzyl ether (XIV), b1 73-5°, n22D 1.5312. A solid residue (8 g.) recrystd. gave 3,4-dichlorobenzyl alc., m. 50-3°. XIV predominated over the alc. XIV was prepared in quant. yield from XIII and NaOEt in alc. by refluxing 10 min. Using the same procedure and the same amts. of reagents as described above, XIII was converted into VI in 25% yield, m. 112°. VI was also oxidized to 3,4-dichlorobenzoic acid. V (1.02 moles) in 391.5 g. IX added dropwise at 110° to 157.6 g. IX gave 83 g. CO2, 85.7 g. alc., 3.5 g. AcH, and 2.5 g. HCl. The residue gave 360.1 g. unchanged IX and 188.1 g. crude dimer. This dimer triturated with hot ligroine gave XI, m. 207-8°. There was obtained along with this solid 10 g. of an oil. Attempts to determine the mol. weight of XI with the Rast camphor method indicated only partial solubility XI (13.5 g.) reduced by the Clemmensen technique with HCl in AcOH gave a nearly quant. yield of di-p-cymene (XIVa), m. 155.5°. XI (5.5 g.) in 500 ml. AcOH, 500 ml. H2O, and 2.5 g. HgCl2 refluxed 3 days with a water trap attached, the H2O and AcOH removed, and the residue taken up in Et2O and extracted with 5% Na2CO3 gave 2,3-bis(p-formylphenyl)-2,3-dimethylbutane (XV), m. 207-8° (95% alc.), also obtained by hydrolysis of XI with HgCl2 in aqueous Me2CO. It was converted by the Wolff-Kishner technique to XIVa, m. 153-4°. KMnO4 in acid solution oxidized XI in poor yield to the corresponding **dicarboxylic** acid (XVI), but produced some terephthalic acid. Better yields (50%) were obtained by digesting small samples of XI in aqueous dioxane with NaOH and adding H2O2 at intervals over a 16-hr. period. The acid was obtained on evaporating the solvent, and acidifying; redissolving in dilute NaOH, reacidifying, and washing gave XVI, m. 280-5° (decomposition), also obtained by oxidizing with H2O2 in AcOH. **Decarboxylation** of XVI gave 2,3-dimethyl-2,3-diphenylbutane. XV was converted into the dihydrazone, m. 275-6°; dioxime m. 215-17°; 2,4-dinitrophenylhydrazone m. 319° (decomposition). I (66.5 g.) in 460 g. IX added dropwise at 130° to 94.7 g. IX gave from the thermal decomposition 39.7 g. CO2, 14.8 g. MeOAc, and 16 l. CH4, 314.6

g. unchanged IX, and 134.2 g. crude X, m. 138° (ligroine). Hydrolysis of X with KOH in aqueous dioxane gave largely resin. In neutral solution the yield of p-(p-isopropyl-ω,ω-dimethylphenacyl)benzaldehyde (XVII) was better, but did not approximate that obtained from hydrolysis in acidic medium. X (18.2 g.) in 800 ml. H2O and 800 ml. AcOH containing 2.5 g. HgCl2 refluxed 96 hrs., the H2O and AcOH removed in vacuo, gave from the remaining red oil 14.9 g. crystals and 0.51 g. resin. The crystals taken up in Et2O, extracted with 5% Na2CO3, and the Et2O evaporated gave X, m. 104°. The Et2O insol. Na salt obtained through the Na2CO3 extraction was acidified to give p-(p-isopropyl-ω,ω-dimethylphenacyl)benzoic acid (XVIII), m. 198°, also obtained by oxidizing XVII with H2O2 in dilute AcOH. The CO group in XVIII was shown by treatment with H2NOH.HCl to give the oxoiminohydroxamic acid, m. 211-12°. XVII gave the dihydrazone, m. 284-5°, dioxime, m. 194-6°, and the 2,4-dinitrophenylhydrazone, m. 205-6°.

IT **6575-24-2**, Acetic acid, (2,6-dichlorophenyl)-
(and phenacyl ester)

RN 6575-24-2 CAPLUS

CN Benzeneacetic acid, 2,6-dichloro- (9CI) (CA INDEX NAME)